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Adding the Environmental TEM to the *In Situ* Toolbox for Catalyst Characterization

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Studies of catalyst materials using *in situ* techniques usually involve a compromise of the *in situ* conditions in order to fulfil the requirements compatible with the instrumentation for characterization. These requirements include sample geometry, temperature, gas environment etc.
Environmental TEM depends on complementary experiments and characterization techniques. Normally, this is done in parallel with experiments separated in time and space [1] or mimicking a reactor bed by changing the feed gas composition according to reactivity and conversion measured in dedicated catalyst set-ups [2]. Furthermore, dedicated transfer holders have been used to transfer catalyst samples between reactor set-ups and TEM at room temperature in inert atmosphere [3].

This work takes the sample transfer concept one step further by use of a dedicated TEM transfer holder that is able to enclose the sample in a gaseous environment at temperatures up to approx. 900°C [4]. This introduces the possible to study specimens over a wider pressure range by combining specimen characterization and treatment performed on different *in situ* set-ups with complementary capabilities.

The performance of catalysts is strongly linked to nanoparticle (NP) dispersion, size, morphology and chemical state during reaction. By use of advanced *in situ* tool box including ETEM, XRD, and XAS a number of novel heterogeneous catalytic systems have been investigated *in situ* during NP formation and catalytic reaction in order to determine the structure-activity relations.

This work shows examples of how the ETEM is used in combination with other *in situ* techniques to elucidate and study and relate performance and local structure of catalysts. Figure 1 shows the methanol yield versus temperature for two novel methanol catalysts based on Ni-Ga and Pd-Ga intermetallic alloys, respectively. The performance is comparable or even higher than the highly optimized $Cu/ZnO/Al_2O_3$ catalyst (at the given conditions), whereas, the CO produced by the reverse water gas shift reaction (rWGS) as a function of temperature is significantly lower than the one given by $Cu/ZnO/Al_2O_3$ catalyst above 200°C. This makes further optimization possible as the rWGS limits the overall methanol yield. Figure 2 shows an ETEM image of a Pd-Ga NP along the [100] zone axis acquired at 550°C in 120 Pa H_2. Furthermore, future possibilities of the *in situ* toolbox will be discussed.

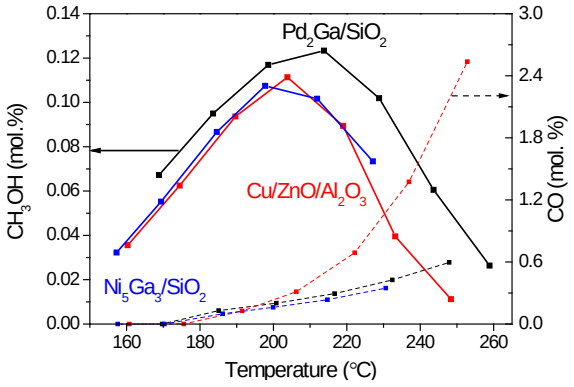

Figure 1. Methanol (solid lines) and CO (dashed lines) yield as a function of reaction temperature.

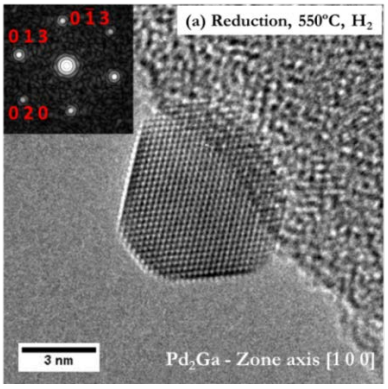

Figure 2. ETEM image of Pd_2Ga NP Acquired at 550°C in 120 Pa H_2.

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